pounds, a more thorough investigation is warranted. It would be of interest to assay the antimicrobial activity of these compounds quantitatively, and also assay the antimicrobial activity of them in commercial products, such as medicinal products, paint films, plastics, and other polymeric materials. The medicinal applications of some of these compounds might prove to be very important. Some of these compounds could serve as plasticizers as well as antimicrobial agents.

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Studies of the Oxygen Bomb Method for Determining Shortening Stabilities

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Abstract

The Oxygen Bomb Method as evolved by the studies presented in this paper gives production and quality control laboratories a procedure by which to measure a fifty-hour shortening in one hour and forty-five minutes. The OBM is twice as precise as in the Active Oxygen Method as shown by the comparison of their two sigma error.

Introduction

THE SEARCH for a faster and more accurate test is a never-ending quest. The measurement of the potential stability of edible shortenings is certainly one of the oil industry's slowest and one whose validity is most questioned. This paper describes a faster and more precise procedure by which to measure stability.

A 50-hr shortening by the 97.8C official AOCS Active Oxygen Method (AOM) (1) may be analyzed in 20 hr by the 110C AOM.

Work has been reported using an Oxygen Bomb Method (OBM) (2–6) to reduce the testing time. One of these reports decreased the analysis time for a 50-hr shortening to two hr and 45 min (4).

This paper describes the work accomplished with an interesting apparatus modification which eliminates the limitations of the oil bath, and cuts the analysis time of a 50-hr shortening to one hr and 45 min. These statistical studies show experimental error effects of temp, pressure, day-to-day variation and sample wt. One study examines the correlation between the OBM and the AOM procedures and compares the standard deviation of both methods.

Dispersants have been used to decrease the testing time and improve the OBM end point determination (3,5,6). For the sake of simplicity we did not use a dispersant. End point determinations were not a problem with our test parameters. Two reports were published (5,6) after the analytical work for this paper had been completed. Pohle et al. (6) used a catalyst to decrease testing time.

Experimental Methods

Apparatus: An 8 in. x 6 in. x 24 in. aluminum block was drilled to accommodate two machined ASTM gasoline gum stability bombs, two thermometers and a thermistor. Two 250-w and one 500-w General Electric strip heaters 24 in. long were screwed into machined grooves on the sides of the alumimum block and connected to a Sargent Model S Thermonitor. The temp of the block and bombs are maintained at $135 \pm 0.1C$ by means of the temp controller with its thermistor being installed into a drilled hole. Three inches of 85% magnesia insulation on the top, sides and bottom of the block aid in maintaining temp. The bombs are fitted with air tire stems to allow a regular tire chuck to be used to fill the bombs with oxygen. The bombs are connected to a Bristol recorder by brass connecting tubes. A rupture disc assembly prevents an undue pressure from harming the recorder.

Procedure. The following procedure is one which evolved from the studies included in this paper.

1) A 10-g sample of shortening is weighed into a glass bomb liner, covered with a glass lid and placed into a heated bomb.

2) The bomb is closed and purged ten times to create a relatively pure oxygen atmosphere. A final pressure of 110 psi oxygen is introduced into the bomb.

3) The filled bomb is placed into the $135 \pm 0.1C$ aluminum block and the recorder started.

The stability time is arbitrarily that time that begins at the insertion of the bomb into the block until

TABLE I
OBM Stability Time (Min) as Affected by Temp

Temp °C	Soy oil Bomb A	Soy oil Bomb B	Fluidized vegetable shortening Bomb A	Fluidized vegetable shortening Bomb B	Plastic mono-glyceride Bomb A	Plastic mono-glyceride Bomb B	Stabilized lard Bomb A	Stabilized lard Bomb B	Mono- and diglyceride emulsified shortening Bomb A	Mono- and diglyceride emulsified shortening Bomb B
125	70	63	163	163	352	352	280	286	164	164
130	45	44	118	125	200	188	200	200	175	173
135	36	35	100	100	139	130	140	135	125	125
140	30	28	63	70	96	94	95	95	82	90
145	23	23	48	48	60	63	60	60	60	65

a two-pound pressure drop from the peak pressure is measured on the recorder.

Results and Discussion

The following studies helped to evolve the final procedure and are an attempt to answer some of the questions that arise at the intorduction of a new procedure.

Effect of Temperature. Five different types of shortening (soy oil, fluidized vegetable shortening, solid shortening emulsified with mono- and diglycerides, stabilized lard, plastic monoglyceride) were tested at five temp (125,130,135,140 and 145C). These tests were run in duplicate on 10-g samples with 100 psi oxygen pressure on two bombs.

Statistically, the difference between bombs was insignificant in this study. Bomb A had an overall average of 120.2 min, while Bomb B's average was 120.0 min. The two sigma testing error of the OBM based on this study was ±6.6 min.

Table I shows a reduction in analysis time of approx 50% for all the shortenings when the temp is raised from 125–135C. While it is believed that the
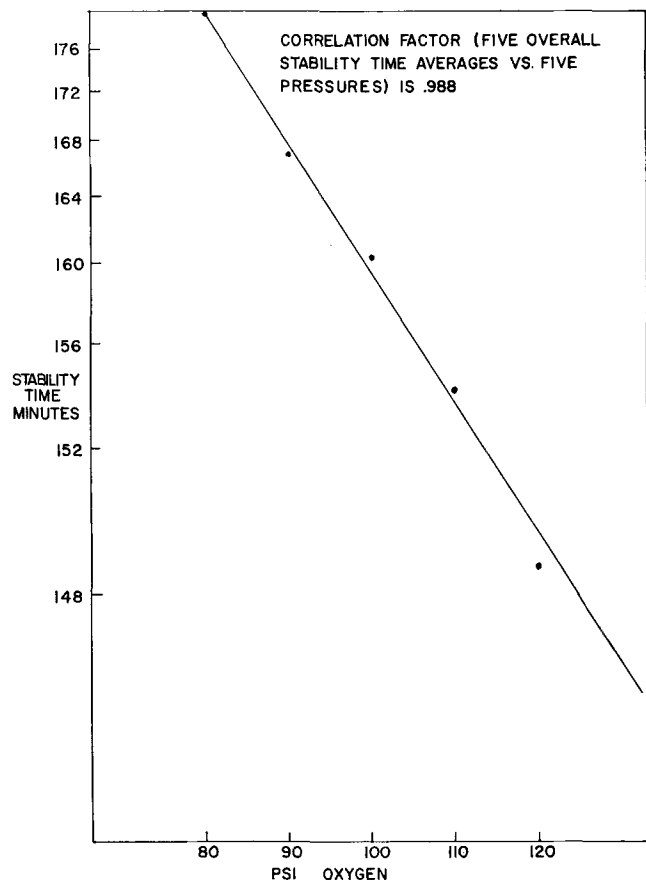

CORRELATION FACTOR (FIVE OVERALL STABILITY TIME AVERAGES VS. FIVE PRESSURES) IS .988

FIG. 2. The average time of Table III's five pressures is plotted against the different pressures on semi-log paper.

TABLE II
Experimental Error Study

Sample I

Day	Bomb A (min) Operator J	Bomb A (min) Operator R	Bomb B (min) Operator J	Bomb B (min) Operator R
1	125	127	125	125
2	125	130	127	124
3	130	135	128	130
4	133	130	123	126

Sample II

Day	Bomb A (min) Operator J	Bomb A (min) Operator R	Bomb B (min) Operator J	Bomb B (min) Operator R
1	133	137	140	137
2	143	140	133	140
3	140	145	140	135
4	140	138	137	137

two sigma error at 125C would be the same as at 135C, it must be realized that a 5-min error at 125C would be much less than at 135C. It was decided, however, that 135C was a reasonable compromise between speed and precision for the largest percentage of shortenings tested.

The temp effects are linear if the temp is plotted against the log of the time in min (Fig. 1). The correlation factor for the temp and the log of stability for the shortenings were relatively good, e.g. mono- and diglyceride emulsified shortening—.998; soy oil—.987; stabilized lard—.998; plastic monoglyceride—.994; fluidized vegetable shortening—.992.

Testing Error. The experimental testing error was determined by two operators running two types of shortening on two bombs on four different days at 135C with 100 psi oxygen.

The statistical analysis of Table II showed the basic two sigma error to be ±5.4 min. The two sigma variation due to bomb differences was estimated at ±3.8 min. The day-to-day variation was responsible for a two sigma of ±4.2 min. If the latter two effects are combined with the basic variation, the estimated total variation of the method is ±7.8 min.

Another estimate of ±6.6 can be gotten by pooling all the data on the two shortening samples and then calculating the two sigma variation. However, the previous estimate of ±7.8 min is one which would be expected during year-in and year-out testing.

Effect of Sample Weight and Oxygen Pressure. Seeking the effects of sample size and oxygen pressure, one operator ran one shortening at five different oxygen pressures (80,90,100,110 and 120 psi) with five sample wt (5,10,15,20 and 25 g) at 135C for a total of 25 randomized tests.

Table III shows that both the sample wt and oxygen pressure significantly affect the results.

When five different oxygen pressures at one sample wt are plotted vs. stability time, the graph appears to be curvilinear. The curves may be straightened by correlating the pressure with the log of the time (Fig. 2). When correlating the log of the five overall stabil-

TABLE III
Effects of Sample Wt and Oxygen Pressure at 135C Expressed in Min

Sample wt Grams	Oxygen pressure (psi) 80 min	90 min	100 min	110 min	120 min	Avg min
5	177	163	155	148	145	157.6
10	175	163	157	155	147	159.4
15	182	167	158	149	147	160.6
20	178	170	168	157	152	165.0
25	185	171	163	161	155	167.0
Avg	179.4	166.8	160.2	154.0	149.2	

TABLE IV

Stabilities of Fifteen Shortenings by the AOM and OBM

Identification	OBM in min	AOM in hr				
		Lab 1	Lab 2	Lab 3	Lab 4	Lab 5
Vegetable						
A	215	118	126	125	120	105
B	168	90	96	133	80	76
C	148	63	69	59	67.5	68
D	401	308	324	348	312	285
E	245	130	154	147	142.5	90
F	315	277	242	290	255	220
G	42	13	10	11	15	11
H	53	25	21	22	25	20
I	90	45	40	42	42.5	40
Animal						
A	146	118	113	133	107.5	38
B	489	476	342	391	470	360
C	36	11	12	10	10	8
D	26	7	6	6	7.5	9
E	83	58	48	68	60	48
F	144	108	104	111	97.5	85

ity time averages with the five pressures, the correlation is .988. Since the curve flattens out as the pressure increases, it was decided to operate at 110 psi in order that minor pressure variations would not adversely affect the results.

OBM Versus AOM Correlation Study. This study included the stability analysis of nine vegetable and six animal shortenings by five different laboratories with the AOM. The OBM series were run in duplicate at 135C with 110 psi oxygen pressure.

In the statistical analysis of Table IV, Lab 5's results were discarded as several of its results were obviously in error. The results of the vegetable series were correlated separately from the animal shortenings as previous work had indicated that separate curves existed for both types.

Assuming the correlation between the AOM and

REGRESSION EQUATION LOG TIME= 6.6384 − (.0334)(TEMP)

CORRELATION FACTOR (TEMP VS. LOG TIME) .998

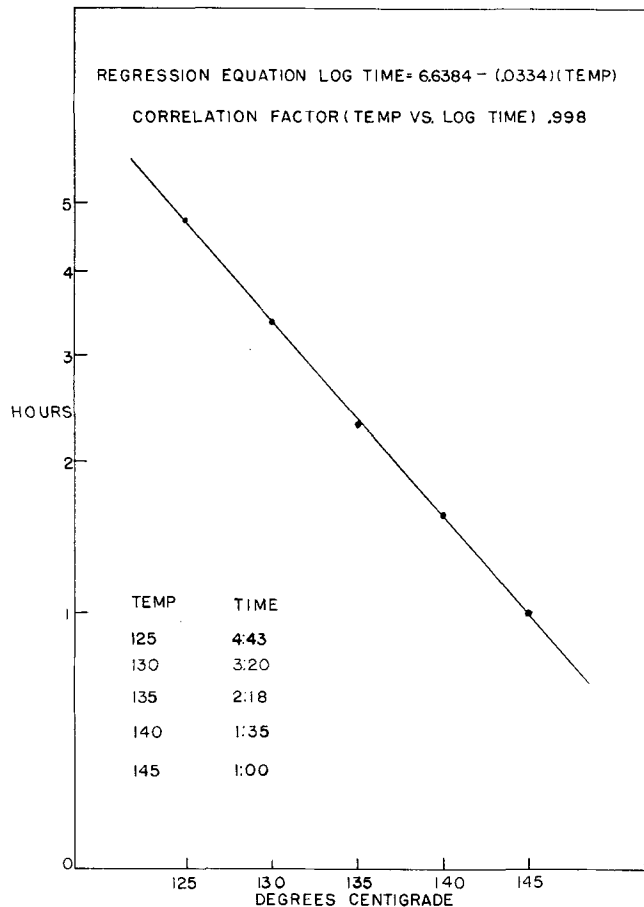| TEMP	TIME
125	4:43
130	3:20
135	2:18
140	1:35
145	1:00

FIG. 1. One lard sample analyzed by the OBM at five different temp. The five temp are plotted against time on semi-log paper.
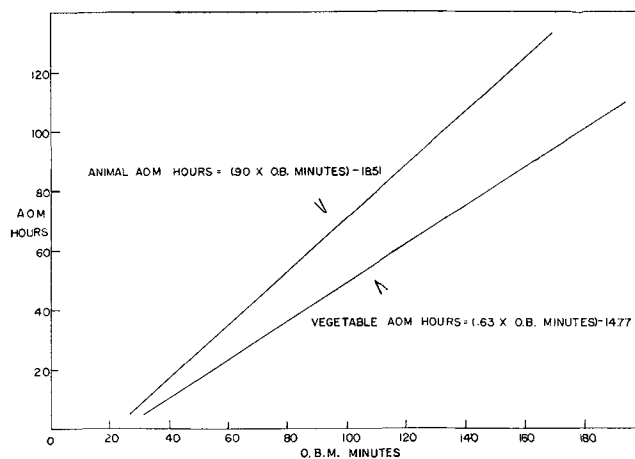

FIG. 3. Correlation of the OBM and AOM for the stability analysis of animal and vegetable shortenings. Correlation coefficients are .988 and .987, respectively.

OBM result was linear, an equation for the straight line regression was calculated. This is a line that estimates the AOM hr from the OBM results. The vegetable shortening results gave a two sigma limit of ±47 AOM hr with a correlation coefficient of .971. However, as shortening with a high stability of over 150 AOM hr causes little concern, the results of the vegetable shortenings D and F were discarded and a second correlation regression analysis was calculated. The second correlation coefficient was .987 and the confidence band was equal to ±15.6 AOM hr. The regression equation for this correlation is:

$$\text{AOM hr} = (.63 \times \text{O.B. min}) - 14.77. \text{ (Fig. 3)}$$

The animal shortening series were similar analyzed. All the data was used at first. The correlation coefficient was .989 and the errors' limits were ±41.6 AOM hr. Since the lower stability samples are of the most concern, the B results were eliminated and the correlation regression was recalculated. The correlation changed to .988 and the two sigma error was reduced to ±15.2 AOM hr. The regression equation for this correlation is:

$$\text{AOM hr} = (.90 \times \text{O.B. min}) - 18.51. \text{ (Fig. 3)}$$

An analysis of variance of the AOM labs excluding Lab 5 gave a two sigma erro of ±34.2 AOM hr.

The 15.2 AOM hr and 34.2 AOM hr described above are estimates of variance covering from zero to 140 AOM hr. These are average estimates of variance represented by parallel confidence bands. These values are too wide for low stability shortenings and too narrow for high stability shortenings. The relationship of the accuracy of the OBM to the accuracy of the AOM will hold at a low or high stability.

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